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[54] Title of Invention: Process for producing gossypol from di-aniline gossypol

[57] Abstract:

The present invention relates to a process for preparing gossypol from di-aniline gossypol, comprising decomplexing di-aniline gossypol at 4-50°C by adding water, acetone, antioxidant and sulfuric acid, crystallizing by adding water and antioxidant, and solid-liquid separating to obtain crude gossypol product while recovering acetone. The present invention has advantages including simply process, low cost, high extraction rate, and high benefit.

What is claimed is:

1. A process for preparing gossypol from di-aniline gossypol, characterized in that a crude gossypol product is obtained via steps comprising decomplexing di-aniline gossypol at 4-50°C by adding water, acetone, antioxidant and sulfuric acid, crystallizing by adding water and antioxidant, and solid-liquid separating to obtain crude gossypol product while recovering acetone.

2. The process for preparing gossypol from di-aniline gossypol according to claim 1, characterized in that the crystallization step is performed under stirring, and the stirring time is from 5 to 180 minutes.

3. The process for preparing gossypol from di-aniline gossypol according to claim 1 or 2, characterized in that the crystallization step is performed under standing, and the standing time is from 20 to 600 minutes.

4. The process for preparing gossypol from di-aniline gossypol according to claim 3, characterized in that the solid-liquid separating step is set between the di-aniline gossypol decomplexation step and the crystallization step, wherein the solid phase is unreacted di-aniline gossypol, and the filtrate enters the crystallization step.

5. The process for preparing gossypol from di-aniline gossypol according to claim 4, characterized in that the crude gossypol product is washed and dried under vacuum to obtain a finished gossypol product, wherein the washing is performed with water, aqueous solution of ethanol and 6# solvent separately.

Process for producing gossypol from di-aniline gossypol

Technical field

The present invention relates to a process for producing gossypol from di-aniline gossypol.

Background Art

The cottonseed blasto slices contain cottonseed oil, proteins and gossypol. At present, the extraction of gossypol is difficult and has poor economic results, and the technique for extraction of gossypol in the prior art produce only di-aniline gossypol. For example, the Chinese Patent CN1040453C discloses a process for removing gossypol from cottonseed kernel slices, wherein the wet gossypol-removed stuff is obtained after extracting gossypol and oil from cottonseed kernels in a circular extraction machine with an ethanol-hexane mixture, and the dried gossypol removed stuff is obtained through a process of desolvation in a combined system of flash airflow desolvation device and a horizontal drier. This process can produce only aniline gossypol, while aniline gossypol has a lower economic benefit in comparison with gossypol, so that the value of cottonseed is not sufficiently utilized.

Contents of the invention

The object of the present invention is to provide a process for preparing gossypol from di-aniline gossypol having advantages including simply process, low cost, high extraction rate, and high benefit.

The key of the present invention lies in converting aniline gossypol into gossypol via steps including decomplexation, crystallization, etc.

The technical solution for carrying out the present invention is as follows:

A process for preparing gossypol from di-aniline gossypol comprises decomplexing di-aniline gossypol at 4-50°C by adding water, acetone, antioxidant and sulfuric acid, crystallizing by adding water and antioxidant, and solid-liquid separating to obtain a crude gossypol product while recovering acetone.

In said process for preparing gossypol from di-aniline gossypol, the crystallization step is performed under stirring, and the stirring time is from 5 to 180 minutes to increase yield.

In said process for preparing gossypol from di-aniline gossypol, the crystallization step is performed under standing, and the standing time is from 20 to 600 minutes to increase yield.

In said process for preparing gossypol from di-aniline gossypol, the solid-liquid separating step is set between the di-aniline gossypol decomplexation step and the crystallization step, wherein the solid phase is unreacted di-aniline gossypol and the filtrate enters the crystallization step.

In said process for preparing gossypol from di-aniline gossypol, the crude gossypol product is washed and dried under vacuum to obtain a finished gossypol product, wherein the washing is performed with water, aqueous solution of ethanol and 6# solvent separately.

In the prior art, gossypol is converted into aniline-gossypol via complexation reaction with aniline. In the present invention, aniline-gossypol is converted into gossypol via a process comprising decomplexation reaction, crystallization, etc., wherein said process has advantages including high extraction rate, simple process, low cost, and high economic benefit, and the solvent can be readily recovered, so that the value of cottonseed is sufficiently utilized.

Brief description of drawings

The present invention is detailedly described in combination with examples as follows.

Figure 1 is the process flow diagram of Example 1 of the present invention.

Examples

Example 1

The decomplexation reaction of di-aniline gossypol (see also Figure 1) is carried out by adding 9 kg water, 891 kg acetone, 1 kg antioxidant sodium sulfite, and 50 kg di-aniline gossypol into a decomplexation vessel, then dropwise adding sulfuric acid under stirring so that the decomplexation reaction is performed at 50°C until the solution becomes clear. The crystallization is carried out by adding 3000 kg water and 1 kg sodium sulfite into a crystallization vessel, then pumping the product of the decomplexation reaction into the crystallization vessel under stirring; after stirring for 180 minutes and standing for 20 minutes, a crude gossypol product is obtained by filtration. Acetone is recovered by

distilling the mother liquor. The crude gossypol product is washed with water, aqueous solution of ethanol and 6# solvent separately, and dried under vacuum to obtain the finished gossypol product.

Example 2

Example 2 differs from Example 1 in that the decomplexation reaction of di-aniline gossypol is carried out by firstly adding 315 kg water, 585 kg acetone, 3 kg antioxidant into the decomplexation vessel and the decomplexation reaction is performed at 5°C; and the crystallization is carried out by firstly adding 2500 kg water and 4 kg antioxidant into the crystallization vessel, the stirring time is 5 minutes, and the standing time is 600 minutes.

Example 3

Example 3 differs from Example 1 in that the decomplexation reaction of di-aniline gossypol is carried out by firstly adding 120 kg water, 780 kg acetone, 2 kg antioxidant into the decomplexation vessel and the decomplexation reaction is performed at 24°C; and the crystallization is carried out by firstly adding 1300 kg water and 2 kg antioxidant into the crystallization vessel, the stirring time is 90 minutes, and the standing time is 400 minutes.

Example 4

Example 4 differs from Example 1 in that the decomplexation reaction of di-aniline gossypol is carried out by firstly adding 30 kg water, 870 kg acetone, 1.1 kg antioxidant into the decomplexation vessel and the decomplexation reaction is performed at 10°C; and the crystallization is carried out by firstly adding 1000 kg water and 2.3 kg antioxidant into the crystallization vessel, the stirring time is 40 minutes, and the standing time is 600 minutes.

Example 5

Example 5 differs from Example 1 in that the decomplexation reaction of di-aniline gossypol is carried out by firstly adding 270 kg water, 630 kg acetone, 2.5 kg antioxidant into the decomplexation vessel and the decomplexation reaction is performed at 40°C; and the crystallization is carried out by firstly adding 800 kg water and 1.5 kg antioxidant into the crystallization vessel, the stirring time is 170 minutes, and the standing time is 60 minutes.

Example 6

Example 6 differs from Example 1 in that a solid-liquid separating step is set between the decomplexation step of di-aniline gossypol and the crystallization step, wherein the solid

phase is unreacted di-aniline gossypol and the filtrate enters the crystallization step.

In the above examples, the crystallization step may comprises one of stirring and standing or none of them, and the solid-liquid separation is one of vacuum filtration, pressure filtration, centrifugal separation, etc.

Figure 1